ASD TR 7-886 (VIII)

ASD INTERIM REPORT 7-886 (VIII)
April, 1963

DEVELOPMENT OF 2400°F FORGING DIE SYSTEM

H. Nudelman
T. Watmough
P. R. Gouwens

ARMOUR RESEARCH FOUNDATION
of
Illinois Institute of Technology

Contract: AF 33(600)-42861
ASD Project: 7-886
ARF Project B220
Interim Technical Progress Report
28 December 1962 - 27 March 1963

The prototype die has been assembled and heating experiments initiated. These indicated that with Calrod heating a die face temperature of 1300°F can be obtained. This will be supplemented by gas flame radiation heating on the die face to attain a temperature of 2400°F. Experiments utilizing calcium flouride-sodium flouride salt combinations to give oxidation protection to the refractory metal die components were successfully performed. A spectrum of viscosities can be obtained with this salt combination by relatively minor adjustments in proportion of either of the components.

BASIC INDUSTRY BRANCH
MANUFACTURING TECHNOLOGY LABORATORY

Aeronautical Systems Command
Air Force Systems Command
United States Air Force
Wright-Patterson Air Force Base, Ohio

ABSTRACT - SUMMARY
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Research continued on the third phase of this program designed to evaluate new materials for use as a 2400° F forging die. The ultimate objective is the creation of a true hot-working technology for refractory metals.

Experiments have been continued to determine the feasibility of using a viscous protection coating rather than the conventional solid coating for the refractory metals. It has been shown that salts composed of sodium flouride and calcium flouride in various proportions give a spectrum of fluidity and viscosities between 1800 and 2400° F, while maintaining oxidation protection to both TZC and P4 refractory metal alloys.

The prototype die system has been assembled and heating experiments performed on the lower die using only Calrod heating. Die face temperature of 1300° F were obtained and supplemental gas flame radiation heating to the die face should allow a temperature of 2400° F to be reached.

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FOREWARD

This Interim Technical Progress Report covers the work performed under Contract AF 33(600)-42861 from 28 December 1962 to 27 March 1963. It is published for technical information only and does not necessarily represent the recommendations, conclusions, or approval of the Air Force.

This contract with Armour Research Foundation of Illinois Institute of Technology, Chicago, Illinois, was initiated under ASD Manufacturing Technology Laboratory Project 7-886, "Development of 2400°F Forging Die System." It is administered under the direction of Mr. George W. Trickett of the Basic Industry Branch, Manufacturing Technology Laboratory, Aeronautical Systems Division, Wright-Patterson Air Force Base, Ohio.

Mr. Paul R. Gouwens is the project director, with Mr. T. Watmough, and Mr. H. Nudelman principally responsible for experimental work on Phases I, II, and III, respectively. Dr. W. Rostoker and Mr. R. J. Van Thyne are serving as internal ARF consultants. All of the above are members of the Foundation's Metals and Ceramics Research Division. This report is designated as ARF-B220-24 by Armour Research Foundation.

The primary objective of the Air Force Manufacturing Methods Program is to increase producibility, and improve the quality and efficiency of fabrication of aircraft, missiles, and components thereof. This report is being disseminated in order that methods and/or equipment development may be used throughout industry, thereby reducing costs and giving "MORE AIR FORCE PER DOLLAR."

Your comments are solicited on the potential utilization of the information contained herein as applied to your present or future production programs. Suggestions concerning additional Manufacturing Methods development required on this or other subjects will be appreciated.

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DEVELOPMENT OF 2400°F FORGING DIE SYSTEM

I. INTRODUCTION

This is the eighth interim technical report, covering the period 28 December 1962 to 27 March 1963 on Contract No. AF 33(600)-42861. This program, an extension of previous Air Force contracts AF 33(600)-35530 and AF 33(600)-35914, is designed to extend the previous development of a high-temperature die system for forging steel, which used a die temperature up to 1600°F.

The ultimate objective of the present research is the forging of refractory metals with dies operating at about 2400°F, but determination of the other limitations of the present hot die system was also necessarily included. The detailed objectives of the present program are to:

1. Evaluate the upper operating temperature limit of forging dies cast from Inconel 713C. Previously, failure did not occur even at 1600°F and a load of 1000 tons.
2. Determine the minimum number of forging steps from unshaped blank to advanced finished shape using the hot die system.
3. Attempt to develop a die material of metallic, ceramic, or composite metal-ceramic structure which can operate at about 2400°F, without atmospheric protection and under loads required to hot-work refractory metals.
4. Devise methods for manufacturing die blocks using the materials developed for 2400°F applications.
5. Produce dies and forge sufficient molybdenum alloy parts to prove the process and materials developed.

Work during this past quarter has been concentrated on the third objective. The first and second phases of the research are now completed.

II. EXPERIMENTAL RESULTS

PHASE III - Development of a Die Material Suitable for Service at 2400°F

A. Selection of Metallic Materials

The criteria for acceptance of a suitable die material have been established as follows:

1. A minimum compressive yield strength of 25,000 psi at 2400°F.
2. Oxidation resistance for 100 hr at 2400°F.

While these standards are quite arbitrary, there is good indication that the strength requirements represent a realistic appraisal of the conditions to be met during forging of molybdenum at 3000°F.

Resistance to oxidation is the major problem and has been approached by a consideration of solid protective coatings and also by renewable viscous protective media. Previous reports document the failure of the solid coatings on various molybdenum alloys. However, to ensure that every avenue to obtain a satisfactory integral coating was explored, a further series of 97% W-3% Mo samples was submitted to Chromalloy Corporation for application of their proprietory coating. After their return to ARF, one coated sample was evaluated for oxidation resistance at 2400°F in dry air. Figure 1 shows the condition of this sample after three hours in such an environment. It can be observed that coating has started to deteriorate, and rounding of corners has occurred. Consequently this would be considered an unsatisfactory oxidation-resistant coating for the criteria previously detailed.

Previous reports indicated that a preliminary evaluation of the possible use of molten salts to protect the refractory metals against oxidation yielded encouraging results. This aspect was further pursued, and samples of TZC and TZM molybdenum-base alloys and P1 and P4 tungsten-base alloys were immersed in a high speed steel heat treatment salt at 2400°F for a 48 hr period. Table I contains the weight loss data for these experiments; the results obtained are most encouraging .

Difficulties with this type of salt, which is composed mainly of barium chloride, are the extremely low viscosity and surface tension of the molten material. In practice this may lead to difficulty in securing adequate protection for the dies, especially if the dies have any great degree of shape complexity. Accordingly, a basic salt combination was sought whose viscosity

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Figure 1. Coated Specimen of P4 Tungsten Alloy After 4 hr at 2400° F in Dry Air

TABLE I

WEIGHT LOSS OF TZC, TZM, P1 AND P4 IN HIGH SPEED HEAT TREATMENT SALT *

Elapsed Time, hr	TZC	TZM	P1	P4
	Weight, gm			
0	6. 8707	16. 9336	13. 9664	11. 0491
24	6. 8898	16. 9213	13. 9803	11. 0028
48	6. 8762	16. 9066	13. 9622	10. 9963

* Designated Liquid Heat 1500 supplied by E. Houghton.

could be varied with temperature, or with a change in the percentage of one of the constituents. This implies that the press forging temperature of 2400°F would be within the liquidus-solidus boundary of the particular salt combination chosen. A literature search through the ceramic phase diagram data showed a small number of possible combinations, which are listed in Table II.

To obtain an approximate idea of the viscosity of possible salt combinations at various temperatures, a series of heating experiments was performed utilizing the sodium fluoride-calcium fluoride combination. Initially three salt mixtures were prepared for a study of their viscosity characteristics. These were as follows:

Combination 1	53% Sodium Fluoride 47% Calcium Fluoride
Combination 2	25% Sodium Fluoride 75% Calcium Fluoride
Combination 3	10% Sodium Fluoride 90% Calcium Fluoride

Each combination was heated in a clay-graphite crucible to 2400°F and its viscosity visually evaluated. Combination 1 was extremely fluid and heavily attacked an Inconel strip suspended in the molten salt. Combination 2 was somewhat more viscous, but it also attacked the Inconel strip. The third combination was extremely viscous at 2400°F with only a slight attack on the suspended Inconel strip noted. It is realized that protection will have to be afforded the refractory metal at temperatures much lower than 2400°F, and the viscosity at these lower temperatures is also important, if a continuous barrier to atmospheric oxygen is to be maintained. A series of experiments to visually evaluate the viscosity of the salt combinations was therefore performed at 1800°F, and the following observations were made:

Combination 1	very fluid and low viscosity
Combination 2	fluid but high viscosity
Combination 3	low fluidity and pasty consistency.

In view of the extreme fluidity of the Combination 1 salts, at both 1800 and 2400°F, it was decided that this particular pair should be excluded from any further consideration, and consequently another combination was

TABLE II

SALT COMBINATIONS WITH FAVORABLE LIQUIDUS-SOLIDUS RANGES

Salt Combination			Percentage of Salt	Liquidus Temperatures, °C
CaF_2	+	NaF	0 NaF* 68 NaF*	1418 810
CaF_2	+	CaF_2	20 Ca F_2* 100 CaF_2*	670 1418
BcF	+	CaF_2	0 Be Fe** 90 Be F**	1418 495
LiF	+	UF_3	30 UF_3* 100 UF_3*	718 1500
NaF	+	UF_3	30 UF_3* 100 UF_3*	700 1500
$2Na_2P_2O_5$	+	$Na_2O\ 2SiO_2$	90 Na_2 O $2SiO_2$** 30 Na_2 O $2SiO_2$**	820 1350
K_2Cl_2	+	$CaSO_4$	30 Ca SO_4* 100 K_2 Cl_2*	687 1450
CaO	+	Cr_2O_3	55 Cr_2 O_3** 35 Cr_2 O_3**	900 1350
CdO	+	B_2O_3	45 B_2 O_3* 100Cd O	800 ?
B_2O_3	+	CoO	63 Co O 90 Co O	900 1350

* Mol. percentage
** Weight percentage

was substituted, namely: 82.5% calcium fluoride and 17.5% sodium fluoride, designated as Combination 4. This combination at 1800° F had low fluidity and high viscosity. Combinations 1, 2, and 4 were evaluated at 2000° F for fluidity and viscosity, and the results were similar to those obtained at 1800° F. The variations in viscosity and fluidity obtained, especially in the case of combinations 2 and 4, are encouraging. Specimens of TZC molybdenum alloy and P4 tungsten alloy were immersed in each salt at 2000° F and 1800° F for 6 hr each and then were visually examined. No loss of edges or oxidation of the specimen was apparent after this treatment. Weight loss data will be obtained during the next report period to confirm the protection offered by the three salt combinations at the various temperatures.

Subsequent to the last Interim Report No. VII, tentative recommendations were made to the Contracting Officer and Technical Monitor concerning the selection of die materials. On the basis of work done to date, two materials were selected for construction and evaluation as an actual forging die. The two candidates were selected from the two major material catagories investigated, i. e., metallic and ceramic. It was recommended that the bottom die impression be carried in a tungsten alloy, P4, containing 85% W and 15% Mo, and that the top die be fabricated from a self-bonded silicon carbide commercially produced as Refrax. By making the dual material selection, a most valid comparison of relative performance should be obtained. At the same time the design of the forged part (a modified half nozzle of a rocket engine) is such that the stress system active on the top die is essentially one of hydrostatic compression during the deformation stroke. This favors the low ratio of tensile to compression stress characteristic of all ceramics by minimizing the possibility of large tensile components of the forging stress.

The bottom die selection of alloyed tungsten was predicated on acceptable high temperature strength, relatively ready availability of simple cast shapes, and the selection of a viscous salt as an oxidation protection medium. The latter can be most advantageously used under conditions where gravity and die configuration will permit and encourage continued contact with the die; thus a gravity-formed salt pool can really be used only on a bottom die unless thin films would prove to adhere to and protect the upper die.

The recommendation was also made that the molybdenum alloy TZM be used as the forging stock during Phase V of the program. All of these recommendations were accepted by means of a telephoned approval, which also authorized proceeding with Phase IV and Phase V research.

B. Prototype Die for Heating Experiments

Several minor machining modifications were carried out to facilitate the assembly of the forging die components. The preparation of the Calrods was completed with the winding of inner and outer coils for the upper and lower die stacks. A series of slots was milled into the stainless steel retainer rings to permit the installation of thermocouples into the prototype die inserts. The lower die stack was assembled, and six thermocouples were attached to the inner and outer areas of the heater blocks directly beneath the lower die surface. Mechanical thermocouple connections were made in which holes were drilled in the heater blocks and peened over after thermocouple insertion. Two additional thermocouples were installed into drilled holes in the prototype die and cemented over. The lower stack was assembled first since this operation was less complex. This approach permitted the development of techniques for installing thermocouples and made disassembly relatively easy.

An angle iron frame was welded together to serve as a heating stand. In this arrangement the upper die stack will be suspended from the top of the frame, while the lower die stack will be supported on a reinforced hydraulic jack within the frame. This will allow the die opening to be adjusted in a manner simulating actual hot forging die practice. After assembly, the lower die stack was installed in place, and a series of heating experiments were performed. Only the Calrod heaters were used for this preliminary work, and the thermocouple emf values were read manually. Current plans call for the use of a 20 point recording potentiometer for advanced heating studies wherein both stacks are in operation.

A 50% duty Calrod cycle provided the heating power for the initial trial. After a 4 hr period the hottest portion, the inner heater block, was 1115°F and the coldest portion, the outer heater block, was 630°F. Die

temperatures of 670 and 680°F were recorded. An increase in the duty cycle to a 60% level for an additional 4 hr raised the general temperature level about 200°F.

In order to improve heating efficiency the area between the outer heater block and the shroud was packed with Fiberfrax, a fibrous mullite material, and the exposed upper stack surface was covered with a 1-in. thick transite plate. A second heating trial was conducted over a two-day period in which a 75% duty cycle was maintained during the day and a 25% duty cycle at night. Temperature equilibrium was obtained after about 20 hr and this provided the following temperature levels:

inner heater block	1560°F
outer heater block	1045°F
prototype die	1145-1290°F

This performance appeared satisfactory, and the assembly of the upper die stack was started. A similar thermocouple arrangement will be used, and in addition two thermocouples will be installed on the surface of the retainer ring at the inner and outer edges. As in the lower stack the space between the shroud and outer heater block will be packed with Fiberfrax to minimize heat losses. After assembly the upper die stack will be mounted on the angle iron frame, and both dies will be given a heating trial using only the Calrod heaters. When this is completed and the results are analyzed, gas burner heating will be added. Six burners, of the type used previously in hot-punch experiments, and the same gas-air mixer will make up the additional heating system. This will permit a realistic appraisal of the final heating system, and is expected to provide information facilitating the final hot forging die design and the selection of optimum operating procedures.

III. SUMMARY

The possibility of using a liquid salt as a medium of protection against oxidation of the refractory metal die components has been further explored. A barium chloride base heat treatment salt successfully inhibited oxidation of TZC, TZM, P1, and P4 samples at 2400°F. However this salt is extremely

fluid at this temperature and could give problems in use in the forging dies. A literature search uncovered a number of possible salt combinations whose systems had liquidus-solidus temperatures within the approximate range of 700 to 1400°F. Preliminary evaluation of the sodium fluoride-calcium fluoride salt combination indicated that a spectrum of fluidity and viscosity is obtainable by relatively minor variations in composition. These salts also appear to provide protection against oxidation of the TZC and P4 alloys.

Assembly of the prototype lower die system has been completed, and breaking experiments have been performed utilizing Calrod heating elements.

IV. FUTURE WORK

It is planned to:

1. Conclude study of salt protection systems.
2. Investigate the heating characteristics and temperature distribution of the complete prototype die assembly.
3. Develop the final design for the complete hot forging die system.

V. LOGBOOKS AND CONTRIBUTING PERSONNEL

Data gathered during the research work are contained in ARF Logbooks C11166, C11167, C11168, C11169, C11908, and C12901. Foundation personnel include:

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Respectfully submitted,

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Project 7-886

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